October 17, 2000

Objective: Evaluate different cleaning procedures for eliminating polydimethyl siloxane (silicone) grease from mild steel coupons.

Approach: Expose steel coupons to Dow Corning high temperature vacuum grease (silicone), and then attempt removal using a variety of cleaning techniques. Analyze the surface for the presence of silicone using secondary ion mass spectrometry (SIMS), which has exceptional sensitivity for detection of silicone surface contaminants.

Coupon Preparation: Twenty-five square coupons were cut from 1/16" hot rolled steel stock. [Details of the coupon preparation is included in the Vacuum Grease Cleaning Test Number 2, 10/11/00 by L. Zirker] The side dimension was ¼". All coupons were smeared with silicone and allowed to stand over night, except for the control samples (1,2,18,19), which were untreated with silicone. Samples 3 – 25 were subjected to cleaning procedures that consisted of wiping with a Kimwipe or Q-Tip, either alone or with a solvent. The cleaning procedure for each sample type is described in Table 1.

Coupon number	Silicone exposure	Cleaning procedure
1-2	No	No solvent, wiped with Kimwipe.
3-5	Yes	Wiped with Methylene chloride ($MeCl_2$) soaked Kimwipe
6-8	Yes	No solvent, wiped with Kimwipe
9-11	Yes	Wiped with Ethanol soaked Kimwipe
12-14	Yes	Wiped with Ethanol and then with acetone soaked Kimwipe
15-17	Yes	Wiped with toluene soaked Kimwipe
18-19	No	Wiped with $MeCl_2$ soaked Kimwipe
20-22	Yes	Soaked in biodegradable soaps and wiped with Kimwipe
23	Yes	Double cleaned/scrubbed with Ethanol using Q-Tips*
24	Yes	Double cleaned/scrubbed with Toluene using Q-Tips. **
25	Yes	Double cleaned/scrubbed with $MeCl_2$ using Q-Tips.***

Table 1. Cleaning procedure for steel coupons analyzed.

*This sample was one of the previously cleaned coupons (9-11) that was re-cleaned and re-tested.

**This sample was one of the previously cleaned coupons (15-17) that was re-cleaned and re-tested.

***This sample was one of the previously cleaned coupons (18-19) that was re-cleaned and re-tested.

SIMS Analyses: Analyses were performed using an in-house built, INEEL triple quadrupole secondary ion mass spectrometer operated in the MS^1 mode. The primary projectile was ReO_4^- operated at 4.5 KeV, at a primary ion current of about 150 picoamperes. Coupons were attached to sample holders using adhesive from double-sided tape.

Results. A typical positive ion SIMS spectrum of an exposed, cleaned coupon contained ions that originated from the silicone (*m/z* 73, 147), and from surface contamination that is always present on steel (*e.g.*, *m/z* 41, 27, 23 and others)(Figure 1). To assess cleaning efficacy, the abundance of the silicone-derived ion at *m/z* 73 was correlated with cleaning procedure. However, when multiple layers of silicone were present on the surface, the absolute abundance of the silicone-derived ions was variable, which made comparison using absolute abundances meaningless. To overcome this problem, the *m/z* 73 ion abundances were ratioed to the abundance of a surface contaminant endogenous to the steel surface, *viz.*, Na^+ at *m/z* 23. This treatment did not result in a quantitative measurement of siloxane surface concentration, but did enable comparison of the different cleaning procedures (Figure 2).

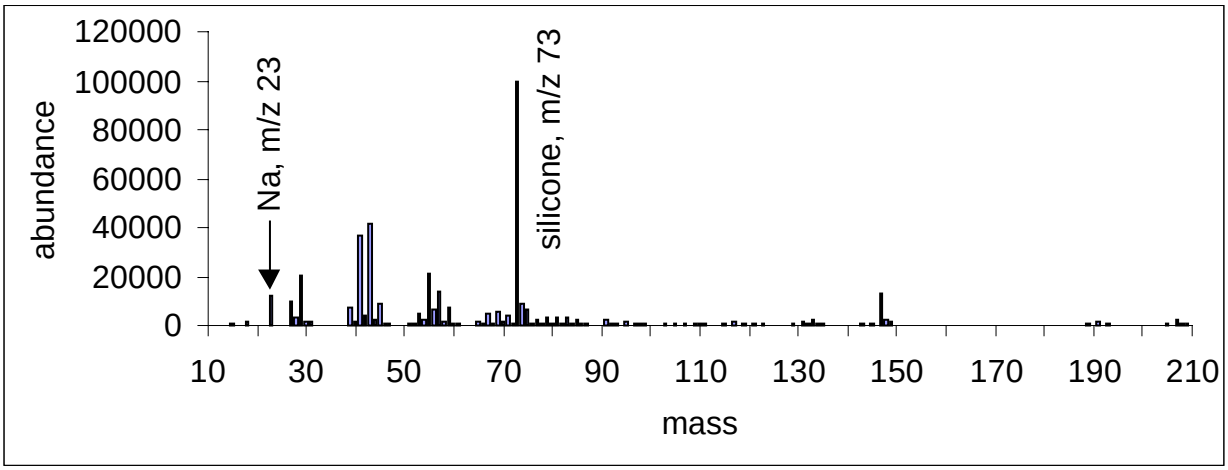

Figure 1. Typical positive ion SIMS spectrum of a steel coupon exposed to silicone, then subjected to cleaning.

Analysis of the untreated coupons (1,2,18, and 19) showed no discernable siloxane contamination.

Three analyses of coupons cleaned only by wiping (no solvent) (6 – 8) resulted in I_{73}/I_{23} values in excess of 300, indicating substantial siloxane contamination.

Utilization of a solvent typically worked better. The order of decreasing effectiveness was toluene > $MeCl_2$ > ethanol (EtOH) + acetone > ethanol. The level of effectiveness of the single toluene cleaning was equaled using a biodegradable soap (coupons 20 – 22).

Scrubbing twice using solvent and a Q-Tip proved to be the most effective treatment (coupons 23-25). The order of solvent effectiveness was toluene ~

$MeCl_2$ > ethanol + acetone. The silicone was still clearly present on the coupon after scrubbing with ethanol, but at much reduced levels.

Without prior calibration research, SIMS is not a quantitative technique, and was not quantitative in these experiments. However, based on previous research, we know that our detection limits can exceed 1/1000 of a molecular layer under the best circumstances. The siloxane polymers are very readily detected using SIMS, and I estimate that the surface concentration of the siloxane does not exceed this level on the coupons double scrubbed using toluene or $MeCl_2$. For the siloxanes, I estimate that 1/1000 of a molecular layer on a flat steel surface equates to about 1 nanogram (1×10^{-9} g) per mm^2.

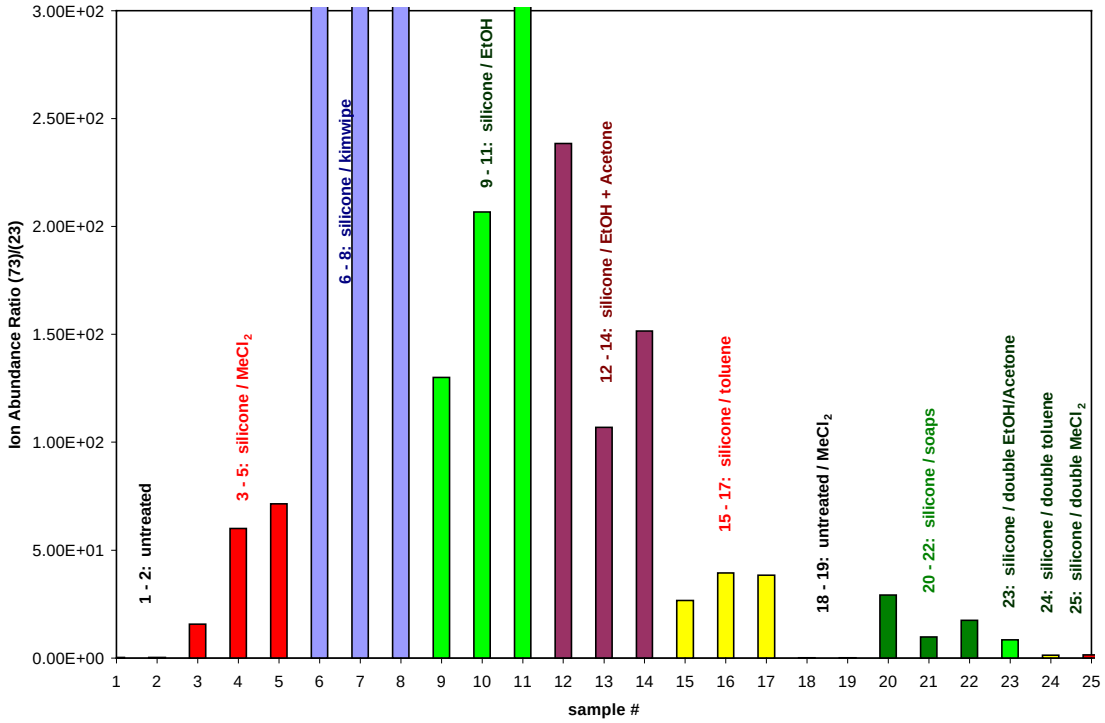

Figure 2. Plot of the ion abundance ratio (I_{73}/I_{23}) versus coupon number.

Conclusions. Comparing the single scrub procedures attempted, utilization of toluene works the best, but still leaves substantial siloxane on the steel surface. A double scrub using toluene or $MeCl_2$ removes nearly all of the siloxane from the steel surface.